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ADHESION BETWEEN ATOMICALLY PURE METALLIC SURFACES

PART IV

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EXPERIMENTAL INVESTIGATIONS OF SOLID ADHESION

Experimental investigation of solid-solid adhesion at Syracuse University is directed toward the three main problem areas cited in the theoretical review submitted on 1 May 1965 to NASA as a semi-annual report (1). Briefly these areas include: a) physical adhesion, i.e. no experimental evidence of attractive forces between clean metal surfaces prior to physical contact have been cited; b) measurement of true contact area during the adhesion process, i.e. no known experimental technique has been developed which will provide true contact area of adhesion; c) effect of adsorbed, or impurity, species on metal-metal adhesion, i.e. no adhesion techniques have been developed to date for surfaces with a measured degree of purity concentration.

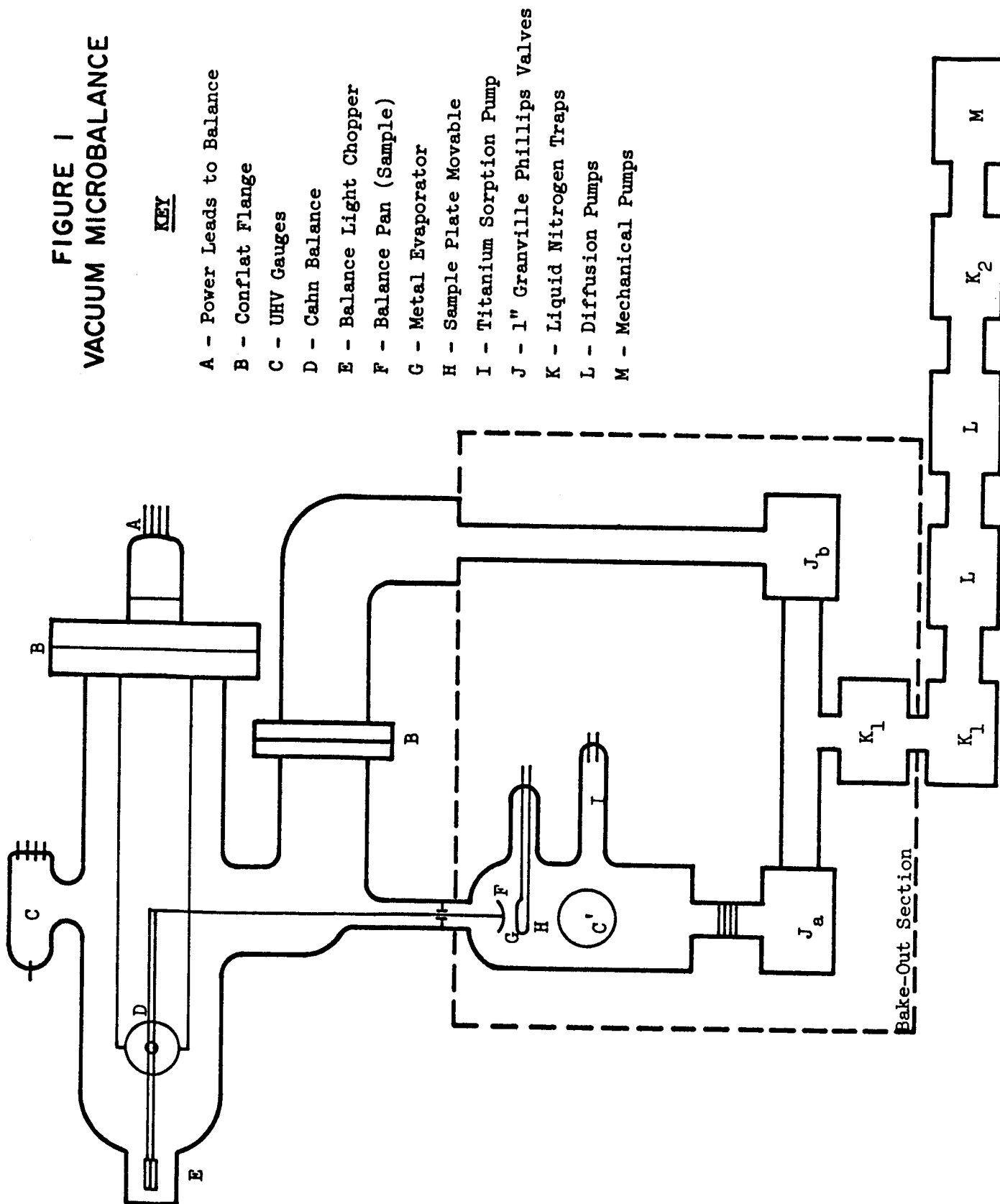The following is a brief progress report of these activities:

Physical Adhesion - As indicated in the previous report (1), physical adhesion is defined as dispersion forces of attraction occurring at separation distances less than one micron which exist between parallel plates prior to physical contact. One may also include in this category forces of attraction between dissimilar metal plates separated by small distances, < 1 μ, which are electrically short-circuited giving rise to an electrostatic field attraction dependent on the contact potential of the two metals. The dispersion and field forces have been studied to a limited extend for quartz and certain metal specimens (in air), and these data are in agreement with the theoretical equations (1) developed to date.

Davis and Engelke (2) proposed that the observed static adhesion force data from the quartz-quartz system, which they investigated, coincided with an extrapolated line of the force of attraction vs distance of separation plot used normally to justify the dispersion force of attraction theory. Since the force theory proposed by Lifshitz (3) is only dependent upon the optical properties of

the material under investigation and since the optical properties of most materials are obtainable over most of the critical wave lengths, the ultimate proof of the proposed theories as well as a confirmation of the experimental observations could well lead to a direct mechanism of estimating the adhesion properties of two unknown materials using only the optical properties of those materials. Furthermore, the general nature of the theoretical equation allows the inclusion of all materials, i.e. inorganic, organic, solid or liquid, rather than specifically metals which is certainly to the advantage of the study.

The system designed and constructed to make physical adhesion measurements between atomically clean metal plates is shown in Figure 1. The apparatus consists of a vacuum micro-balance to which is attached one metal sample plate, or receptor for a metal vapor film. The balance is a Cahn RG automatic electrobalance capable of sensitivities to 0.1 μgrams and produces a DC output proportional to the balance load which is recorded on a 10 millivolt electronic recorder. Changes in apparent mass of the sample plate (F) due to forces of attraction between it and the movable sample plate (H) thus may be recorded. The movable sample plate (H) consists of an aluminum substrate supported on a short section of 0.007" x 0.187" bi-metallic strip which acts as a resistor in a high current circuit. By varying the current through the strip, e.g. varying its temperatures, the movable sample plate can be made to move at a linear rate (calibrated beforehand) into contact with the sample plate on the balance. During progression of the movable plate toward the balance plate variation in the forces on that plate will be recorded as an apparent mass change. Since both the rate of closure, plate-plate, and the rate of mass change are simultaneously known, a correlation between separation distance and attractive force may be determined.

The vacuum loop system illustrated in Figure 1 appeared to be the only solution for obtaining recorded microgram measurements in a static ultra-high

FIGURE 1
VACUUM MICROBALANCE

KEY

A – Power Leads to Balance
B – Conflat Flange
C – UHV Gauges
D – Cahn Balance
E – Balance Light Chopper
F – Balance Pan (Sample)
G – Metal Evaporator
H – Sample Plate Movable
I – Titanium Sorption Pump
J – 1" Granville Phillips Valves
K – Liquid Nitrogen Traps
L – Diffusion Pumps
M – Mechanical Pumps

Bake-Out Section

vacuum (UHV) system, pressures below 10^{-10} Torr, since the balance itself constitutes a primary gas source well above 10^{-8} Torr. Overdesigned pumping systems and slightly modified Cahn balances have been operated to pressures in the 10^{-10} Torr range (as claimed by the producers); however, since the proposed experiments were to include atomically clean and control contaminated surfaces it was felt that the elimination of all possible internal contamination sources, ever present in dynamic vacuum systems, ought to be eliminated. The loop design consists of a balance cell and a test cell which may be isolated from the other through valves, J_a and J_b, since the balance wire passes from one cell to the other through a low conductivity bypass. During the 450°C bakeout cycle of the test cell both valves, J_a and J_b, are open. The balance system pressure is recorded on the hot ion gauge (C). Then after bakeout, valve J_a is closed and the titanium sublimation pump (I) is used to establish the lower pressure in the test cell, while the main pumping system maintains as low a pressure as possible in the balance chamber (10^{-8} Torr estimated). The pumping system consists of two 2" Granville-Phillips liquid nitrogen pumps, two 2" CVC oil diffusion pumps and a Welch duo-seal mechanical pump.

Upon establishment of the pressure in the test cell a fresh film of aluminum is deposited on the sample plates and the movable plate is set into motion. The attractive force is then recorded.

The materials to be tested in this apparatus include various combinations of similar and dissimilar atomically clean metals and the effect of various contaminating gases. The apparatus is in the final stages of assembly. The vacuum system, including the cells, have undergone tests to pressures below 10^{-10} Torr without the presence of the balance mechanism. The balance mechanism was standardized and mounted; however, due to difficulties the balance had to be removed for repairs at the Cahn Company. The repairs having been completed, remounting and recalibration are expected to be accomplished within a month or so.
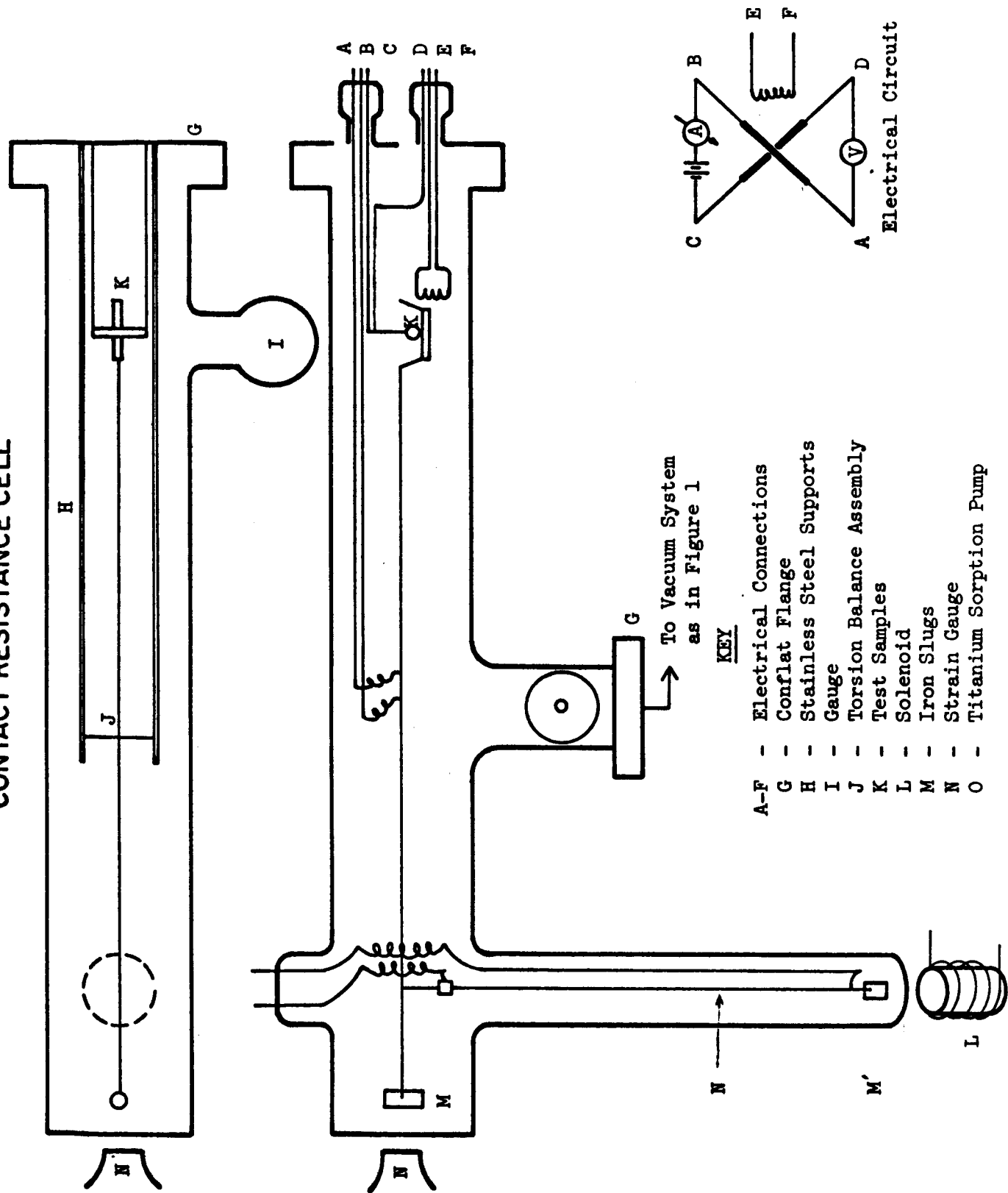
Contact Area Measurements - The discussions in the previous report indicated that

the very nature of static chemical adhesion, i.e. variations from a welded junction to a non-welded junction, was grossly dependent on the atomic species actually present on the surface irrespective of the nature of the bulk material constituting the substrate. The direct observations and limitations of this statement are only qualitative in nature; and if a substantial mechanism of adhesion is to be proposed, quantitative data must be available in its support. At this time most solid-solid adhesion investigations have been directed toward go - no-go information between various materials under such widely varying vacuum and surface conditions that a realistic analysis of the output data appears to be impossible. Direct quantitative data are still required to establish the effects of monolayers and contaminants on the force of adhesion. The main problem in obtaining these quantitative data is the lack of a reliable technique to estimate the amount of surface contaminant at the instant a force measurement is in progress. When these data become available their correlation will be invaluable in the prediction of possible lubricating mechanisms to prevent bulk adhesion.

A system has been studied in this laboratory which seems to show great promise in this direction. Numerous investigators (4,6) in the past have shown that the presence or absence of an adsorbed layer between two metals in contact has a large effect on the contact resistance of that system; furthermore, these data seem to be related to the true area of contact between the specimens. Since the previous investigators were not concerned directly with the precision force measurements nor the perfection of the surface, experimental techniques are lacking; however, the system designed and tested in this laboratory allows for static force measurements below a 5 gm load and can readily be coupled with contact resistance measurements at the junction. The system illustrated schematically in Figure 2 will be used to examine these relationships in several refractory metal systems.

The apparatus consists of a torsion balance with a 23 cm quartz beam mounted on a tungsten torsion wire which is, in turn, supported on two 3/16" stainless

FIGURE 2
CONTACT RESISTANCE CELL

KEY

A-F – Electrical Connections
G – Conflat Flange
H – Stainless Steel Supports
I – Gauge
J – Torsion Balance Assembly
K – Test Samples
L – Solenoid
M – Iron Slugs
N – Strain Gauge
O – Titanium Sorption Pump

To Vacuum System
as in Figure 1

Electrical Circuit

steel support rods (H) welded to a conflat blank-off flange. Of the two metal samples AB and CD, one set (CD) is fixed through insulator supports to the conflat base plate, while the other set is mounted on one arm of the balance beam. The samples are connected electrically to outside of the cell. Thus the electrical circuit shown in Figure 2 represents the test system.
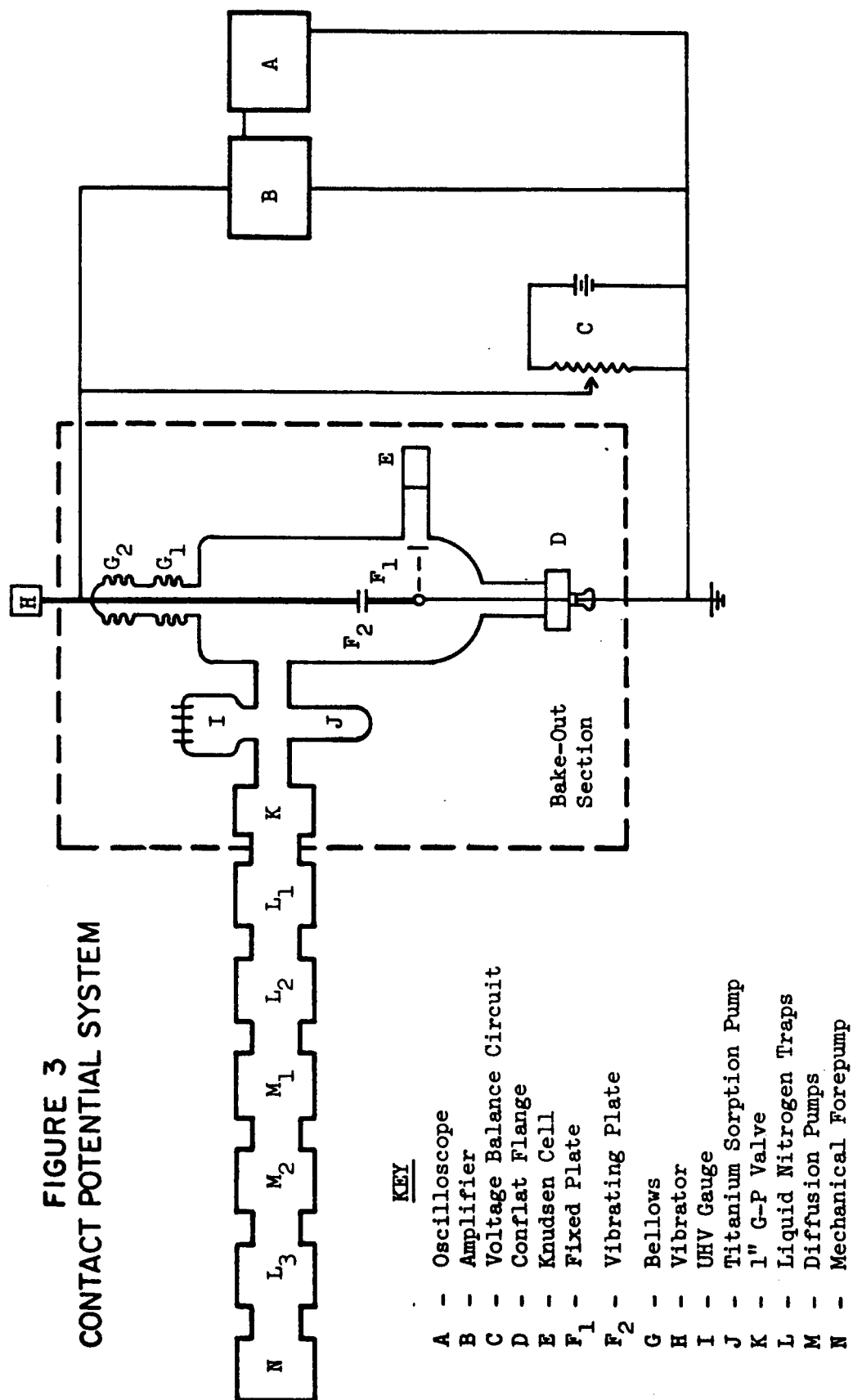
The torsion balance is not actually used as a balance device, in that during a test the beam is held in a fixed force field supplied by the permanent magnet at the end of the 2" pyrex housing tube interacting with the iron core at the beam end. This is called the "positioning magnet" in that it is used to align the samples to the desired point of contact, yet at a separation distance of about 0.1 cm. Sample contact and force measurements are accomplished by increasing the current in solenoid (L) which, in turn, causes an attractive force between it and the iron slug (M') fixed to the end of the strain gauge wire (N). This force is required to shear the attractive force between the iron slug (M) fixed to the balance beam and the positioning magnet. The 0.00095" x 5" straight wire constantan strain gauge (N) is attached to, but electrically insulated from the balance beam, and has a residual resistance of about 80 ohms. Therefore, the force required to bring the samples into contact, i.e. the shearing force of the fixed magnet field, is recorded as a strain. Simultaneous observation of the current-voltage properties in the crossed metal samples indicates the point of physical contact; thereafter, force versus contact resistance properties are recorded as the contact force is increased by means of the external solenoid. After the maximum contact force is achieved, the contact force is reduced (still recording contact resistances) to the point of contact separation. Since the torsion system required a force to shear the fixed magnetic field and this field is superimposed constantly on the contact system, the force of adhesion, or the breaking force, can be measured as the change in zero point at separation. The balance system as described can produce a maximum load of about 5 gms with a

sensitivity of about 10 milligrams. A precision Kelvin bridge utilizing voltages in the range of 6 millivolts is used to detect the resistance values in the system. A Keithley nanovoltmeter (10^{-9} volts) in conjunction with a 30 Ω resistor is used to detect the null point in the bridge circuit.

Modifications in the beam support mechanism are in the process of completion, and are expected to be complete within a month. The molybdenum-molybdenum system will be investigated in order to compare these data with those of the molybdenum-copper (immiscible) system.

Surface Impurities - As indicated in the previous section, the existence of unknown contaminants on a surface to be engaged in an adhesion study may have a gross effect on the results. It is hoped that the contact resistance technique will provide one means of detecting the presence of these layers during a test, thus enabling an investigator to cite a point of reference in describing the surface under investigation. The purpose of the third area of investigation is to examine the utilization of contact potential measurements as a second method of surface characterization prior to adhesion measurements. Since the measurements of the contact potential of a metal or alloy is a very simple procedure yet extremely sensitive to surface condition (7,9), the technique seemed most worthy to consider.

The contact potential apparatus shown in Figure 3 consists of an ultra-high vacuum system (K-N) utilized to evacuate a 5" O.D. x 8" test cell in which a polycrystalline tungsten plate, standard (F_2), is supported parallel to a movable test sample plate (F_1). Cyclic variation of the position of the tungsten plate normal to the test sample plate produces an AC signal due to the contact potential field between the two plates which may be detected on an ocilloscope. If a known DC voltage is supplied to the plates which is opposite, but equal to the contact potential, the observed AC current will disappear; and, the impressed DC potential is equal to, and recorded as, the observed contact potential. The

FIGURE 3
CONTACT POTENTIAL SYSTEM

Bake-Out Section

A, B, C, D, E, F_1, F_2, G, H, I, J, K, L, M, N

G_2 G_1

KEY

A — Oscilloscope
B — Amplifier
C — Voltage Balance Circuit
D — Conflat Flange
E — Knudsen Cell
F_1 — Fixed Plate
F_2 — Vibrating Plate
G — Bellows
H — Vibrator
I — UHV Gauge
J — Titanium Sorption Pump
K — 1" G-P Valve
L — Liquid Nitrogen Traps
M — Diffusion Pumps
N — Mechanical Forepump

use of this technique in conjunction with adhesion studies will be examined after the standardization of the apparatus.

This apparatus is in the early stages of construction and is expected to be in operation within a month or so.

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